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Evaluation of properties of anti-adhesive coatings on the surface of injection moulds made of Al alloys

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ABSTRACT

Purpose: The aim of the research was to determine the most favourable fluoropolymer coatings, deposited on the surface of aluminium moulds, which will be used to share improvements in automotive sectors.

Design/methodology/approach: The paper presents the results of the coatings thickness and surface roughness measurements, AFM analysis, contact angle measurements and surface free energy calculation, wear test and adhesion force measurement of three fluoropolymer coatings.

Findings: On the basis of the investigation, it can be stated that the properties of the tested coating and their adhesion to the aluminium substrate are different. Based on the obtained results it can be concluded that the coating named Coat_134 was characterized by the optimum properties - well anti-adhesion properties and good mechanical properties.

Research limitations/implications: Coating must be applied on the surface of injection tools, with complex shape and wide range of dimensions.

Practical implications: Injection moulds for polyurethane foam, reduction of release agent consumption.

Originality/value: The paper presents comparative research of fluoropolymer coatings in order to determinate adhesion to polyurethane foam with low density.

Keywords: Fluoropolymer coatings, Abrasive wear, Layer thickness, Adhesion force

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PROPERTIES

1. Introduction

The automotive industry produces many elements made from polyurethane that ensure the comfort of using a car. The most exemplary application of polyurethane foams are car seats, without which there would be no question of any travel comfort. Furthermore, there are many other elements that are made of polyurethane foams, many of them are not noticed by the user because they perform specific functions such as car soundproofing or touch comfort. The higher class of the car, the more such elements are existing in it. The parts made of polyurethane foam are made by using injection moulding technology. Moulds are made of aluminium, depending on the production capabilities they are castings or machined blocks. During the process of shaping polyurethane products, the most common production problem is the adhesion of products to the mould, which directly affects a large number of non-compliant products. Of the currently used release agents, the most commonly used is liquid wax. This solution requires manual application on the tool before each injection, significantly extending the production cycle time and increasing costs. The harmfulness of this solution to the health of the operator working in such environment is also significant. In order to overcome the use of the release agent, it was decided to investigate the influence of fluoropolymer coatings on the functional properties of injection moulds. Three test coatings were made in cooperation with the company "Coatresa" located in Barbera del Valles in Spain.

2. Materials and methodology

The subject of the research was fluoropolymer coatings, which were deposited on the surface of the aluminium plates with chemical composition given in Table 1.

Table 1.
Chemical compositions of material

Elements						
Al	Si	Fe	Cu	Mn	Mg	Cr
94.74	0.27	0.035	0.06	0.48	4.28	0.07

The surface of the aluminium plates with an edge length of 150 mm and 50 mm width, were coated with fluoropolymer coatings by electrostatic coating method. Afterwards samples with the coatings were subjected to the heat treating – the coating were cured at temperature T = 400°C. Three coatings were selected from Coatresa's portfolio, whose commercial names are; Coat_152, Coat_138 and Coat_134. According to their marketing data, all coatings have good anti-adhesive properties, resistance to chemicals and low surface porosity [1-8].

2.1. Coatings thickness measurements

The coating thickness measurements were performed using "Ultrameter" thickness gauge type AB400 by Metrison Company with a measuring precision ± 1 μm (for 0-1999 μm measuring ranges). Few measurements were made for each of the tested samples with coatings – calculations of the mean thickness value of the coatings were carried out with excluding the extreme values.

2.2. Surface roughness measurements

The surface roughness measurements were carried by the contact method using a Surftronic 25 (Taylor Hobson) profilometer. The measurements length was 4 mm and the measurements accuracy was ± 0.1 mm. Based on the measurement the characteristic surface roughness parameters were determined: R_a – arithmetical average height (μm) and R_z – average maximum height of the profile (μm).

2.3. AFM analysis

Additionally, in order to determine the surface topography Atomic Force Microscopy (AFM) analysis using an XE-100 by Park System were carried out. The measurements were performed in non-contact mode. The surface roughness parameters: a Rough Mean Square RMS/R_q (μm), the arithmetical average height R_a (μm) and sum of maximum height and maximum depth ΔZ (μm) was calculated over scan area 25 x 25 μm.

2.4. Contact angle measurements and Surface Free Energy calculation

In order to determine the physicochemical properties the samples in initial state and samples after surface modification, the contact angle measurements and Surface Free Energy calculation were performed. The test stand included a Surftronic Universal goniometer by OEG Company and PC with Surface 4.5 software, which was used to analyses to recorded drop image. The sessile drop method was used to obtain the values of the contact angle. The measurements were performed with the use two measure liquids: distilled water (θw) (Poch S.A.) and diiodonomethane (θd) (Merck Sp. z o.o) and drops of these liquids, each 1 µl in volume were applied on the surface of the tested samples. The duration of one measurement was 60 s. The measurements were carried out at standard temperature T = 289 K. The values for free surface energy (SFE) and their polar and dispersion properties for the Owens – Wendt method were given in Table 2. In Owens-Wendt methods were used Formulas 1 and 2.

Table 2.
The values of SFE and their polar and apolar components for measure liquids used in Owens-Wendt method

	Distilled water	Diiodomethane
$\gamma_{L,}$ mJ/m^2	72.80	50.80
γ_L^d, mJ/m^2	21.80	50.80
γ_L^P, mJ/m^2	51.00	0.00

$$\gamma_S = \gamma_S^d + \gamma_S^p \qquad (1)$$

$$\gamma_S(1 + cos\theta) = 2\sqrt{\gamma_S^d \cdot \gamma_L^d} + 2\sqrt{\gamma_S^p \cdot \gamma_L^p} \qquad (2)$$

2.5. Adhesion test

In order to determine the adhesion force between coatings and polyurethane foam the measurements using Hegewal&Pesche testing machine were performed. Foam were injected onto a plate with coating using a sample mould with internal dimensions of 300 mm length, 50 mm width and 10 mm height. Before injection, 50mm of the length of the sample coating is covered by polypropylene foil as well as injection mould. After the foam structure were formed then the mould was removed, a sample of the coating with a polyurethane foam layer was obtained with adhesive layer of 100 mm length and 50 mm width. The free short end of the sample surface is fastened in the upper clamping jaws of the tensile testing machine in such a way that the foam above adhesion area from the test sample is turned by 180 degrees and left hanging over the adhesive section (Fig. 1). The distance between the upper and lower clamping jaws were 200 mm and the pulling speed was 250 mm/minute. Seven tests were carried out and the mean of the results obtained.

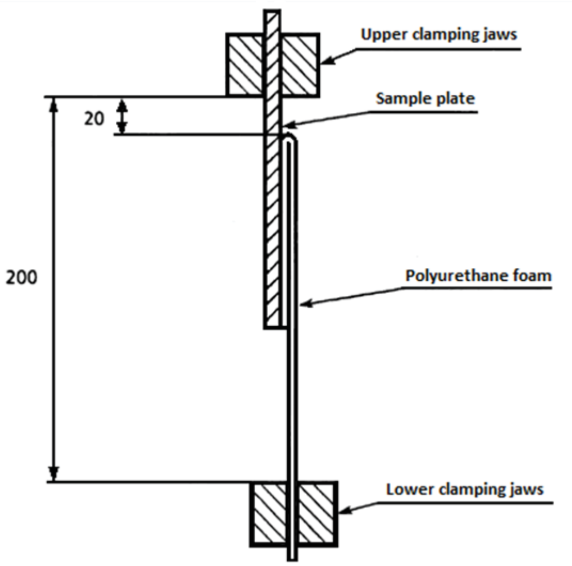

Fig. 1. Scheme of adhesion testing of the coating surface to polyurethane

2.6. Wear test

In order to determine the abrasive wear of tested coatings the Pin-on-disc test using CSM tribometer, equipped with moving stage, an arm with pin holder and a controller were performed. The tested samples were used as plates and the counter specimen consisted of a stainless steel ball (6 mm diameter). Sliding occurs between a stationary pin and a rotating disc with the tested samples. The tests were performed under a constant load of 15 N and the linear speed of 5 cm/s. During the measurements, the values of coefficient of friction (µ) were measured. In addition, the depth and width of the wear tracks were measured using profilometer.

3. Results

3.1. Coatings thickness measurements

The obtained results of coatings thickness measurement were presented in Table 3. The lowest value of layer thickness was recorded for the 1st group of the samples (Coat_134) and the mean value was t = 54.7 ± 2.4. µm. In the other two cases, the similar values of layer thickness were recorded. The mean values for the 2nd (Coat_138) and 3rd (Coat_152) samples group were respectively t = 117.1 ± 5.1 µm and t = 116.9 ± 6.4 µm.

Table 3.
Results of layer thickness measurements

No.	Code	Thickness, µm
1.	Coat_134	54.7 ± 2.4
2.	Coat_138	117.1 ± 5.1
3.	Coat_152	116.9 ± 6.4

3.2. Surface roughness measurements

The results of coatings roughness measurements were presented in the Figure 2. The lowest values of roughness parameters were recorded for the 3rd samples group (Coat_152) and mean values were R_a = 0.05 ± 0.01 µm and R_z = 0.22 ± 0.02 µm, which pointed to 12th grade number of roughness (according to PN-EN ISO 1302:2004). While, for other two tested samples with coatings, higher values of roughness parameters were obtained. The highest values were measured for the 2nd (Coat_138) samples group, which the mean values were an approximately R_a = 0.56 ± 0.08 µm and R_z = 2.51 ± 0.23 µm (8th grade number of roughness). For 1st groups of samples (Coat_134), the recorded values (R_a = 0.14 ± 0.01 µm and R_z = 0.54 ± 0.02 µm) indicate the 10th grade of roughness.

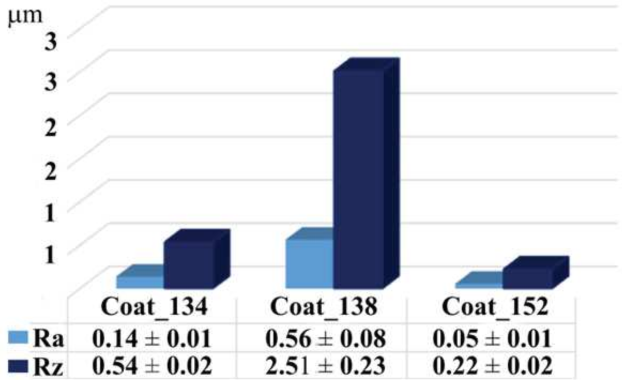

Fig. 2. Results of surface roughness measurements

3.3. AFM analysis

Results of the observation using the Atomic Force Microscopy for all tested samples were presented in Figures 3-5, while the characteristic values describing surface roughness (for micro-areas) were given in Table 4. The lowest values of roughness parameters (R_a = 0.03 µm, R_q = 0.04 µm and ΔZ_{max} = 0.059 µm) and the smallest surface development were recorded for the Coat_152 samples group (Fig. 5).

Table 4.
Results of AFM analysis

No	Code	R_a, µm	R_q (RMS), µm	ΔZ_{max}, µm
1.	Coat_134	0.08	0.10	0.316
2.	Coat_138	0.16	0.20	2.57
3.	Coat_152	0.03	0.04	0.059

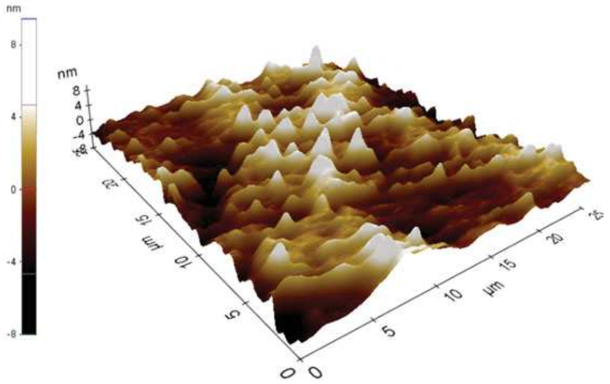

Fig. 3. AFM results of Coat_134, 25 x 25 µm

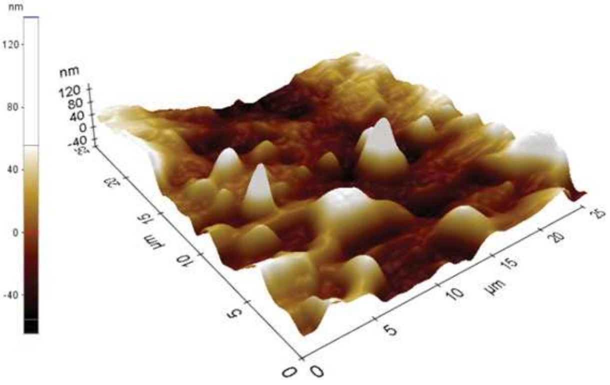

Fig. 4. AFM results of Coat_138, 25 x 25 µm

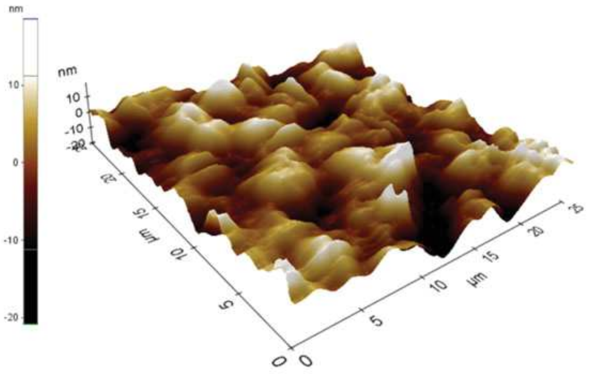

Fig. 5. AFM results of Coat_152, 25 x 25 µm

3.4. Contact angle measurements and Surface Free Energy calculation

The results of contact angle measurements Θ and surface free energy SFE calculation were given in Table 5. For the samples in initial state (pure aluminium surface) the lowest values of contact angle were recorded and the mean value was approximately Θ = 55 ± 6.6°, which indicate the hydrophilic character of the surface. While for the other groups of tested samples after surface modification the hydrophobic character of the surface were observed – obtained values of contact angle were more than 90°. The higher value Θ = 105.8 ± 1.1° was recorded for the 2nd (Coat_152) samples group. For all tested samples except of 1st group, the values of Surface Free Energy SFE were comparable and the mean values were in the range γ_s=9-14 mJ/m^2. Additionally, the SFE calculation shows that for these samples groups, the high values of apolar components and low values of the polar ones were recorded, which indicate to greater affinity of the surface to the apolar group than to the polar ones. The dependence reversal was recorded for samples of 1st group. Based on the obtained results it can be concluded, that with deposition of layer on the tested samples surface, the wettability decreased.

Table 5.
Results of contact angle Θ and Surface Free Energy SFE

No.	Code	Contact angle, °		Surface energy, mJ/m^2		
		Distilled water	Diiodomethane	γ_s	γ_s^d	γ_s^p
1	Aluminium surface	53.1 ± 6.6	55.6 ± 1.8	46.7	14.2	32.5
2.	Coat_134	99.3 ± 2.1	77.0 ± 3.2	19.1	16.4	2.7
3.	Coat_138	95.9 ± 2.4	78.6 ± 2.7	18.6	14.1	4.5
4.	Coat_152	105.8 ± 1.1	92.3 ± 5.9	12.0	8.9	3.1

3.5. Adhesion test

The obtained values of F_{max} and F_{av} were given in Figure 6, and the diagrams, which presents relationship between adhesion force and the displacement of clamping jaws (Fig. 1) were presented in Figures 7-9. The lowest adhesion between the coatings and the polyurethane foam, were recorded for the Coat_152 samples group, which is proved by the values of individual parameters determined through the performed measurements – the mean value of maximum adhesion force F_{max} and average adhesion force F_{av} were respectively F_{max} = 1.6 ± 1.0 N and F_{av} = 1.3 ± 1.0 N. Additionally, it should be noted, that for this samples group, the values of both parameters were comparable. The similar relationship was obtained for the Coat_134 samples group, where the values were F_{max} = 2.0 ± 0.9 N and F_{av} = 1.9 ± 0.7 N. The highest adhesion to the polyurethane foam is exhibited by coating Coat_138F_{max} = 7.6 ± 3.0 N and F_{av} = 3.9 ± 1.0 N.

3.6. Wear test

The results of abrasive wear test were given in Figure 10. According to the record in the safety data sheets, all tested coatings show good abrasion resistance. Based on the obtained results it can be consulted, that the lowest values of friction coefficient were recorded for the Coat_134 and the mean value was an approximately μ = 0.74 ± 0.09. For other two coatings tested, the values of friction coefficient were lowest, and were in the range μ = 0.17 ± 0.05. In Table 6, values of width and depth of abrasion and the values of wear volume (V_f). The highest values of wear volume were obtained for the Coat_134 V_f = 36956 ± 1087 μm, which was characterized by the larges values of friction coefficient. In the case of Coat_138, the degree of abrasion after the wear test was so small that it was not possible to measure the wipe profile. For Coat_158 the mean value of wear volume was V_f = 8850 ± 529 μm.

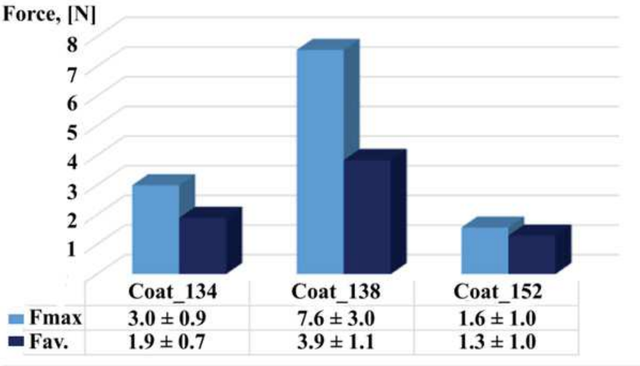

Fig. 6. Results of adhesion test

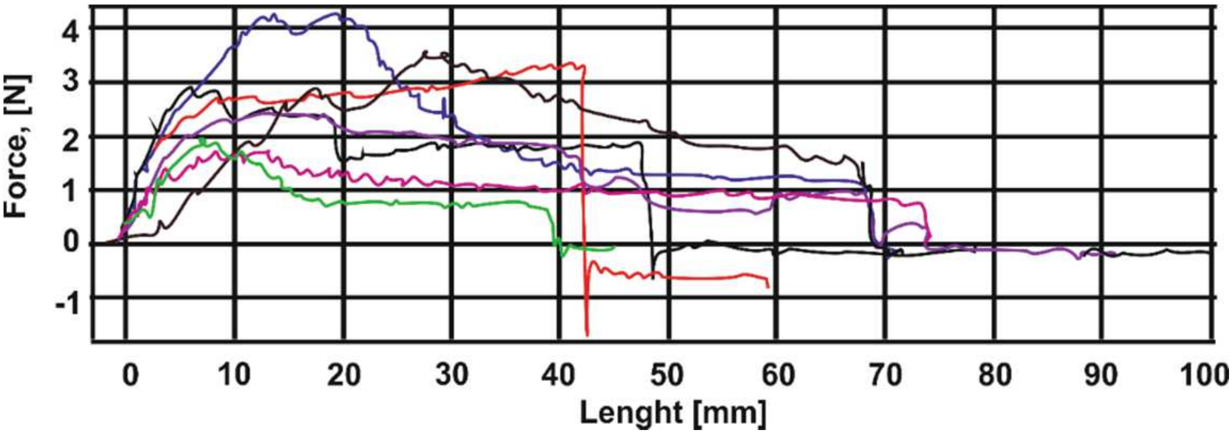

Fig. 7. Coat_134 – relationship between adhesion force and length

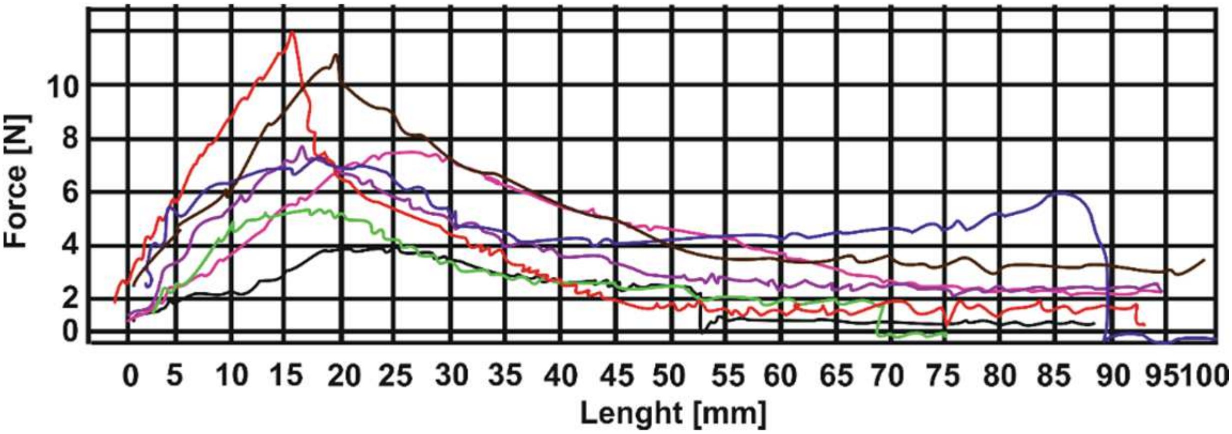

Fig. 8. Coat_138 – relationship between adhesion force and length

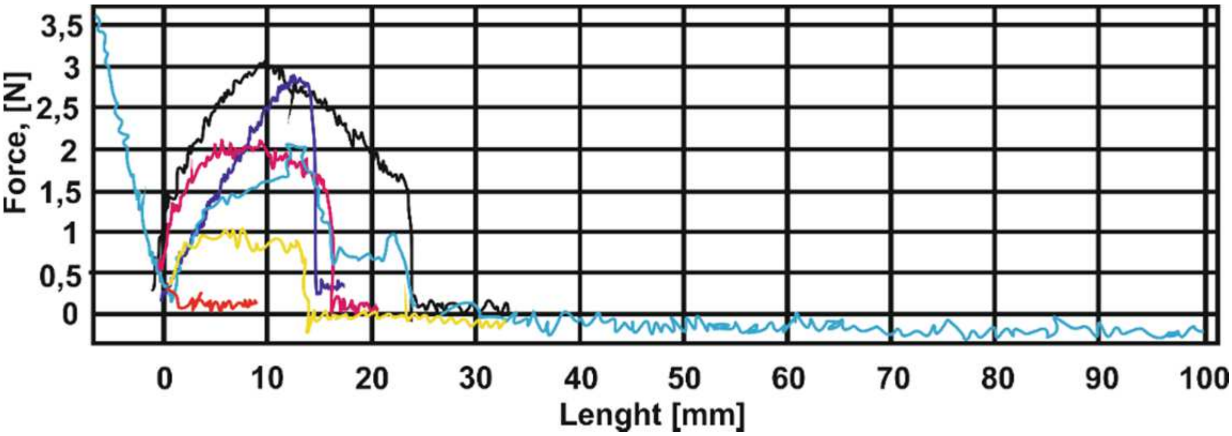

Fig. 9. Coat_152 – relationship between adhesion force and length

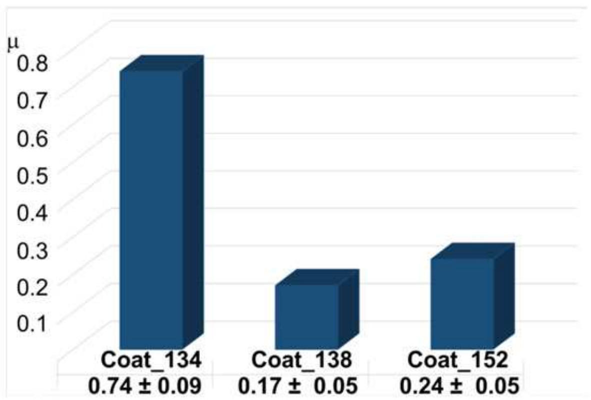

Fig. 10. Results of Pin-on-Plate test

Table 6.
Width and depth of abrasion

	Width, mm	Depth µm	Wear volume (V_f), µm
Coat_134	1.5 ± 0.1	47.1 ± 1.1	36956 ± 1087
Coat_138	-	-	-
Caot_152	0.8 ± 0.5	15.5 ± 2.3	8850 ± 529

4. Conclusions

In the case of the tested coatings, one of the most important properties is their durability. The expected life span of the elements on the surfaces of which the tested coatings are applied is 7 years, which is a standard life time of the project in the automotive industry. During this period of time, it is very difficult to implement any modifications or improvements due to serial production loops, especially without knowledge of the customer. Significant factors affecting the choice of a specific surface modification solution are also the market availability of the coating and the ease of its application. Based on the obtained results it can be concluded, that the coating Coat_152 was characterized by the optimum properties. The coating Coat_152 was characterized by lowest adhesion force and highest wear resistance – the value of force needed to detach the polyurethane foam from the coating being tested is relatively small (F_{max} = 1.6 ± 1.0 N and F_{av} = 1.3 ± 1.0 N). Roughness of the surface also gives the best result from tested coatings Ra = 0,05 measured by profilometer and Ra = 0.03 measured by AFM is related to very high quality of the surface and low porosity. The difference between two measurements might be caused by different areas where measure occurred. Additionally, the obtained values of contact angle measurements (105.8° ± 1.1°) and surface free energy calculated (γ_s = 12.0 mJ/m^2) lead to the hydrophobic character of this coating.

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